CONCLUSIONS AND FUTURE WORK

0.1 Conclusions

The objective of this work was to gain a better understanding of how the design freedom afforded by precise net shape processing techniques such as additive manufacturing might be better utilized to achieve brittle composites with higher toughness. This focus centered how designed structures can affect fracture behavior, what kind of material contrast is needed to achieve higher toughness, and how anisotropic structures might be used to improve toughness beyond what is possible with conventional processing techniques. This increase in toughness would be of particular benefit in the context of ceramics systems, which show promise in structural and engine applications due to their thermal and chemical resilience, but are limited by their brittle failure behavior.

Firstly, we used stable crack growth experiments to understand the influence of designed anisotropy on crack propagation behavior. In particular we explored parameterized composite structures under surfing load conditions with full-field displacement measurement techniques. This analysis showed, in particular, the potential of elastic contrast as a tool for pinning cracks and how a balance between inclusion spacing and inclusion size can maximize the toughness to be well above that of the homogeneous case. Directional toughness showed particular potential for cases of biased or loading because toughness values could be achieved that were similar to those of isotropic materials, but at significantly lower inclusion volume fractions. This improved toughness using smaller volume fractions makes anisotropic inclusions favorable for directional toughness with better retention of bulk matrix properties, which is desirable in structural ceramics.

The effect of anisotropy on both nucleation and propagation was explored through asymmetric void structures called “fracture diodes". These structures used designs of triangular voids to produce a favorable propagation direction, and nucleation was controlled though the presence of an edge notch in the unidirectional cases and larger void at the center of the specimen in the bidirectional case. With this added understanding, a “true diode" design was developed that used rounded triangles and carefully controlled void spacing to further enhance toughness asymmetry, resulting in controlled directional failure 100% of the time. This demonstrates that with a careful control of both compliance and anisotropy, brittle fracture can be controlled in the context of both nucleation as well as propagation of a macrocrack.

After exploring both nucleation and propagation in brittle polymers, the focus transitioned to toughening through designed anisotropy in ceramics. This was achieved using wedge splitting of muscovite mica, which allows for globally stable crack growth. Building on the work of Obreimoff, the behavior of heterogeneous mica sheets with designed, step-wise thickness heterogeneities was investigated. In mica prepared with thickness heterogeneities, a dramatic increase in required separation force occurred when the mica splitting front encountered the thickness increase in the mica sheet. This force enhancement is associated with a change in flexural rigidity of the cleaved mica sheet, which is nonlinear, and the increase in force observed is significantly larger than the splitting force required for the homogeneous constituents. This nonlinearity implies that not only do changes in compliance have an impact on achievable toughness, but variations in the magnitude of compliance change can lead to anisotropy in fracture directions. This anisotropy could be beneficial for maximizing toughness increase and minimizing unstable crack growth due to rapid propagation. This phenomenon could prove to be of significant benefit in layered ceramic composites, as it demonstrates that the introduction of additional stiff layers within compliant regions might further increase effective toughness.

The exploration of ceramics was then extended to systems produced printed preceramic polymer. The investigation explored not only the potential for designed structures as a mechanism to control failure behavior, but also the influence of complex geometry on the mechanical properties of printed ceramics. Four different truss design (two Kelvin cell designs, one octet design, and one mixed design) were characterized both at the structure level through uniaxial compression and at the beam element level using a previously established beam flexure method. Despite attempts to control both size and stiffness based on prior studies of printed preceramic polymer, mechanical analysis of the truss structures after pyrolysis revealed that each of the designs had different strength, stiffness, and shrinkage. Analysis of bulk structures showed a relatively linear scaling between strength and porosity, whereas of the individual beam elements showed a reverse trend from the structures, with the most slender octet beams being the strongest despite the octet structure being weakest. Much of this difference in strength was attributable to size effects arising from the dramatically reduced surface area of the octet beam elements compared to the Kelvin ones. This study shows that when fabricating complex geometries, careful consideration must be given to the structure-dependent shrinkage behavior of additively manufactured ceramics, and the current standards of linear shrinkage and mass loss analysis fail to capture these shrinkage effects. If additive manufacturing of ceramics is to be viable for industrial applications, it is critical to understand these shrinkage behaviors, which will require investigations well beyond those of simple representative bodies. However, if the degree of shrinkage can be managed, there is potential to create truss structures with uniform elastic behavior and controlled failure mechanisms, where crack nucleation and propagation would be dictated by the location of low strength truss elements within the system.

Finally, this work demonstrates how improved processing control not only changes the available design space for composite toughening mechanisms, but it also explores how this new design space can be used to achieve toughening behaviors that have not been well explored. Directional toughening, which is achievable through anisotropy, can produce comparable effective toughness values to isotropic inclusions in one direction, but at a significantly lower volume fraction of inclusion phase, which is beneficial for preserving desirable matrix properties. Furthermore, this directionality can be used to constrain and control crack growth, even before the crack has nucleated. This opens the potential for structures that can be designed to provide toughening based on a particular known crack location, which is dictated by inclusion design and arrangement. Although ceramic additive manufacturing of ceramics is at present limited in terms of bulk structures that can be readily produced, the same anisotropic toughening principles can be applied to truss systems as well. Instead of different materials, different truss elements can be used to control the potential crack nucleation and propagation directions, which opens up further possibilities for increased material toughness.

0.2 Future Work

This exploratory nature of this work means there are a multitude of potential avenues for future work extending off the approaches used in this investigation. The potential areas of research that can emerge from this work will be addressed based on the order of the studies presented.

0.2.1 Propagation Studies Using Surfing Load

Although the surfing load experiments could not be readily extended to explore ceramic systems due the significant stiffness increase, there is still significant potential for the surfing load in the context of exploration of possible two-dimensional toughening designs. The stable crack growth that can be achieved both numerically as well as experimentally means that inclusion arrangements can be readily explored in simulation and then validated in experiment, although the materials suitable for testing would be limited to brittle polymers with lower stiffness than ceramics. This has particular promise because the thin specimens tested in surfing load keep specimen failure constrained to two dimensions, which removes some of the more challenging, hard-to-model aspects of three-dimensional fracture from a numerical standpoint, such as out-of-plane crack twisting and crack bowing. Still, within this two dimensional space, there is significant possibility for the exploration of inclusion arrangements to maximize toughness in a singular direction while minimizing inclusion volume fraction, or to maximize toughness anisotropy, that is, the difference in toughness between different propagation directions. Furthermore, any potential designs could readily be tested experimentally to verify their performance in physical material systems. Some of the experimental limitations with the current surfing load setup would have to be addressed, such as the unwanted buildup of load due to crack pinning. However, it may be possible to address some of this through a redesign of the rail. For example, changing the rail from aluminum to brass to minimize friction and galling and also changing the pin and bushing fittings to have tighter tolerances may make the load buildup more reliable and consistent. With these improvements, it may be possible to refine the diverging section of the rail to be less abrupt, which may help prevent an excess buildup of tension when the crack is pinned at inclusion/matrix interfaces. A refined rail design would also allow for the exploration of additional polymer systems, such as acrylic polymers that can be laser cut. Coupling these with acrylate photopolymers and photosensitive slurries would allow for the testing of multi-material composite designs under surfing load conditions, which would remove the issue associated with crack pinning due to geometric effects, where the crack is forced to bow outward to account for the changes in thickness in the printed specimens in surfing load studies.

0.2.2 Nucleation and Propagation Studies with Fracture Diodes

Experiments with fracture diodes showed the potential of asymmetrical voids to control both the nucleation and subsequent propagation direction of cracks under unbiased loads. However, the exploration of this degree of control was relatively limited both in the context of possible inclusion designs and orientations as well as possible materials of interest. In the context of inclusion designs, all designs in this study were constrained to a single axis, and that axis was chosen to be orthogonal to the loading axis. However, there is significant potential to explore axes that are not orthogonal to the load axis as a means to introduce mixed mode failure behavior into the system as shown in Figure 0.1. This type of mixed mode failure may have

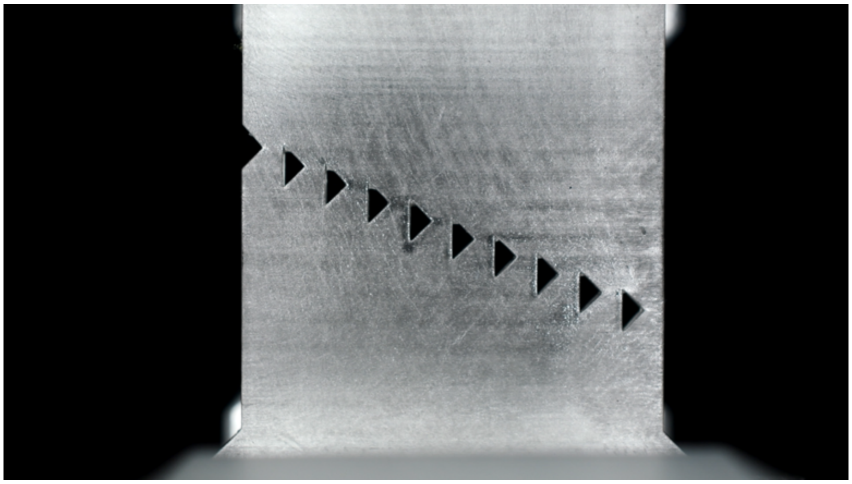


Figure 0.1: Example of fracture diode specimen with inclusion position along an axis not orthogonal to the load axis.

some potential in the context of exploring how different arrangements of porosity can affect failure behavior as well as how crack deflection can be used as a tool to prevent macroscopic failure. Some basic experiments with slanted diodes were performed by C.M. Long, but the extent of designs explored was relatively limited, and additional criteria need to be established in the context of what behaviors are favorable, which would require additional numerical simulations.

Additionally, given that fracture diodes are able to provide such a high degree of failure control, it is logical to explore how this failure control might be exploited to achieve higher toughness behavior. The approach here would be of particular interest because it would deviate slightly from the ideologies of traditional composite toughening. Instead of creating a uniform micro- and macrostructure to prevent macrocrack growth wherever it may occur, the idea would be to instead create locations where macrocracks are more likely to form, and then construct toughening mechanisms specifically around each of these potential locations. In this sense, the fracture diodes could be used to create predictable crack propagation over a short distance, which could then be arrested by much more elaborate toughening mechanisms than can be produced in traditional composites. In this sense, the formation of these macrocracks can be used to relieve stress on other parts of the system, where these more complex toughening mechanisms are not present. This approach is particularly favorable from a numerical standpoint because it is far easier to explore possible toughening mechanisms numerically when the exact location of the macroscopic crack is already known, but these can be difficult to implement practically because the trajectory of the crack is not known. This type of toughening around a known crack location has even been explored relatively recently in the context of machine learning.[1] In these scenarios, the trajectory control afforded by diodes may prove useful by making fracture more predictable.

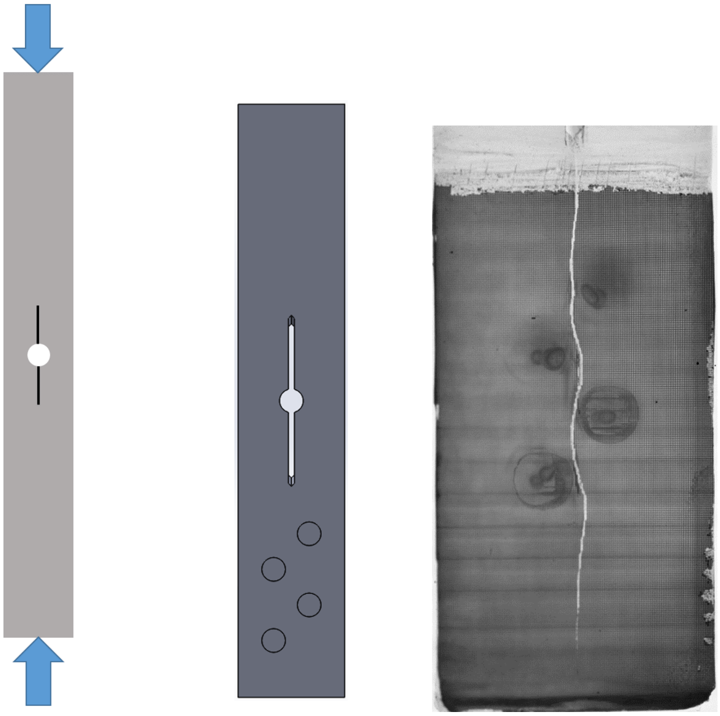
Finally, fracture diode behavior can also be explored in the context of different material systems. Theoretically, this degree of fracture control should also be achievable in stiffer systems like printed ceramics, but this has never been explored in any meaningful detail. In the case of ceramics, higher stiffness will likely result in a larger buildup of elastic energy for a given displacement, so failure may occur more rapidly and catastrophically, which will demand more faster image capture techniques, but the directionality associated with the failure should still be present. If this degree of control could also be achieved in ceramics, there is potential for more complex structures to provide crack arrest and further enhance ceramic toughness, which is a desirable property in many technical ceramics.

0.2.3 Wedge Splitting of Heterogeneous Mica

Compared to the surfing load and fracture diode experiments, the possibilities for further exploring heterogeneous toughening through mica splitting are somewhat limited. The most promising avenues for additional exploration involve either larger sections of uniform mica material or the introduction additional phases. If larger pieces of mica could be obtained, the nonlinearity in the toughening increase due compliance contrast could be explored in greater detail, and structures containing both thickness increases and subsequent thickness decreases could be explored to both maximize the load buildup due to the crack arrest at increases in thickness and minimize the load drop that occurs at decreases in thickness. Along these same lines, the introduction of additional phases to change stiffness contrast becomes much easier when mica sheets are larger in size.

0.2.4 Ceramics from Printed Preceramic Polymers

Although the experiments in this work were somewhat limited due to previously undocumented structurally dependent shrinkage behavior, the potential for additional toughening mechanisms through 3D printed ceramics is still rather large. In the context of truss systems, if shrinkage can be properly accounted for, the potential to use different truss structures with equivalent stiffnesses but different mechanisms (bending vs. stretching), and therefore different failure strengths, presents promise as a way to control the nucleation of cracks in more complex structures. Structurally dependent shrinkage is expected to be present in most ceramic printing techniques that involve a diffusion-mediated conversion process, and controlling this will take some effort, but it is likely achievable with enough data on shrinkage behavior for different print systems. All that would be required to get these data would be a change in shrinkage reporting methodologies in the ceramic additive manufacturing community.



(a) DCDC schematic (b) DCDC design with inclusions (c) Crack-inclusion interaction

Figure 0.2: Images showing a schematic of the DCDC test (a), a DCDC design containing inclusions (b), and the crack-inclusion interactions in a test of a printed photopolymer DCDC specimen (c).

Going beyond truss structures, however, there is also potential for testing of solid ceramic structures if the test chosen such that stable crack growth can be achieved and specimen volume does not have to be large. One promising test is double cleavage drilled compression (DCDC), which involves the compression of slender ceramic or glass specimens with a hole in the center of the specimen, such that the crack nucleates at this hole and grows parallel to the axis of compression, as shown in Figure 0.2a.[2, 3] This test provides stable crack growth on specimens that are relative small in both width and thickness, so it may prove to be a suitable means of evaluating the effect of anisotropic inclusions on ceramic structures. C.M. Long has already done some preliminary tests to explore the potential of DCDC in printed polymer systems, as shown in Figure 0.2b and 0.2c, and crack behavior similar to the response seen in surfing load was seen in DCDC systems, though characterization of toughness proved challenging due to large compressive deformations before fracture. To explore the potential of DCDC for characterizing ceramic composites, SiOC ceramics developed and printed at HRL laboratories are being evaluated in DCDC, and crack length and load are being used to calculate toughness in systems during fracture. It is proposed that the introduction of additional soft inclusions into these SiOC systems, through either changes in thickness or a second phase, would create crack deflection and pinning due to elastic contrast, which should improve macroscopic toughness.

REFERENCES

[1] Grace X. Gu, Chun Teh Chen, and Markus J. Buehler. De novo composite design based on machine learning algorithm. *Extreme Mechanics Letters*, 2018. ISSN 23524316. doi: 10.1016/j.eml.2017.10.001.

[2] C. Janssen. Specimen for fracture mechanics studies on glass. *Proceedings of the 10th International Congress On Glass*, pages 10.23–10.30, 1974.

[3] M. Y. He, M. R. Turner, and A. G. Evans. Analysis of the double cleavage drilled compression specimen for interface fracture energy measurements over a range of mode mixities. *Acta Metallurgica Et Materialia*, 43(9):3453–3458, 1995. ISSN 09567151. doi: 10.1016/0956-7151(95)00036-U.